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CRACK AND GROWTH PREVENTING ADDITIVES IN COMPOSITION 1

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ABSTRACT: A number of specific additives were incorporated into 5"x 8" cylindrical casts of Composition B in order to study: (1) their ability to reduce irreversible growth in the explosive, and, (2) their crack-preventing properties. Results indicate that ortho-nitrophenol is superior, in the above respects, to those additives generally used and reported in the literature.

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Evaluation of the effective functioning of the continuous rod guided missile warhead has raised the possibility that: 1) a high energy jet would develop due to a crack in the explosive and result in the fracture of the continuous rod at detonation and, 2) undue strain, or pressure, in the warhead casing, caused by the explosive growing irreversibly, could result in defective firing. As a consequence, an experimental program was set up, authorized by Bureau of Ordnance Task Assignment NO 301-664/43006/12040 to investigate preventive and remedial measures.

In conformity with the above, the present work relates to the investigation of a number of chemical compounds which, when incorporated in small percentages in large size casts of Comp B, would help to prevent or reduce the amount of cracking and irreversible growth in the explosive.

The present work relates to Part 15 of Key Problem 2.5 on Air Defense Systems Research, as well as to problem 7.2, as given by the Explosives Research Department, NAVORD Report 3906.

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By direction

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CRACK AND GROWTH PREVENTING ADDITIVES IN COMPOSITION B

INTRODUCTION

Two problems associated with Composition B, which are closely interrelated, are irreversible growth and cracking. Attempts made by various activities to eliminate these problems have not been generally successful. The solution to these problems has been approached from many angles, some of which are: (1) the introduction of various types of waxes in the explosive in the hope of discovering some beneficial effect (a); (2) the incorporation in Comp B of various additives that would lower its modulus of elasticity, which condition appeared to impart some desirable anti-cracking properties to the cast explosive (b); (3) the incorporation in Comp B of additives which would form high melting eutectics with TNT - the theory being that a high melting eutectic would prevent growth and a low melting one would do the opposite (c), (d), (f). These, as well as other approaches, have always been only partially successful, when not definitely unsuccessful.

It has been the experience of this investigator that:

1) An additive may appear promising when used in connection with a small cast, but will break down completely when applied to a large size sample - say a 5"x8", or larger, cylindrical cast. Anthracene, for example, was found to do just that, when used as an anti-cracking agent (See TABLE I).

2) An additive may help to reduce, or eliminate, one problem but may do nothing for a second or, indeed, may actually create or promote a second problem, for instance: (a) the additive ortho-nitrotoluene, or cyclohexanone, in Comp B, will help to reduce cracking but will also promote growth and exudation; (b) 2, 4-dinitroanisole causes little or no exudation but is not a good anti-cracking agent, and may actually promote growth; (c) 2, 4-dinitrotoluene is a good anti-growth agent for Comp B, but causes early cracking and crumbling of the cast, etc.

3) Further, what may be good for one type of cast explosive may not be good for another; which vagary extends even to the same type of explosive if the degree of purity should vary from one time to the next, i.e. 2,4,6-trinitroanisole is a good crack preventing agent when used in connection

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with purified TNT but becomes useless if used with commercial TNT.

The work reported herein was undertaken to test two things: (1) the efficacy, as anti-cracking and anti-growth agents, of a small number of additives selected from a total of about 120 agents reported in the literature and, (2) the efficacy, as anti-cracking and anti-growth agents, of two compounds, namely, para-methoxybenzyl alcohol and ortho-nitrophenol, not found in the literature but picked for appearing to be promising experimental material. The compound ortho-nitrophenol was especially chosen because it contains three features which appeared desirable, namely, the presence of the hydroxyl group and the nitro group, together, and an optimum melting point.

EXPERIMENTAL:

Commercial Comp B (7.5 Kgms.) was melted and kept under vacuum (7-9 mm.) for one half hour, after which time it was brought back to atmospheric pressure and 0.5% of additive was introduced with a minimum of stirring. The melt (82-83°C.) was poured into cylindrical steel molds (5" in diameter and 8" in height), preheated to 600-650°C. A steam-jacketed cylindrical riser (3" in diameter and 12" in height) was placed on top of the mold and filled to about three inches from the top with the same melt. Steam was supplied to the jacket as needed. After four hours, at which time the cast had solidified, the riser was removed and the cast allowed to continue cooling at room temperature. This procedure was repeated for every additive herein reported, in addition to one control charge which contained no additive at all (See TABLE I).

Each charge, or cast, was then sawed into three pieces giving round cakes, or slices, (5" in diameter and 2.5" in height). (Eight casts, which were already badly cracked and disintegrated at this point, were discarded. See first eight entries in TABLE I). A slice, taken from each of the remaining eleven casts, went to form one of three groups - A, B, and C -, and each group was then treated differently, as follows: Group A was placed under cracked ice over-night then allowed to come to room temperature - a change of temperature from 0°C. to 24°C. - after which the slices were X-rayed and inspected. This procedure was repeated a second time. The explosive slices then received a third treatment by freezing in ice over-night then suddenly placing them in an oven at 55°-60°C. The results of these three treatments are shown in columns III, IV, and V of TABLE I. It will be noted that while the results are not expressed in a quantitative manner, the properties of the additives, as anti-cracking agents, clearly begin to reveal themselves. A slightly more quantitative

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evaluation of the more promising additives is given in the last column of TABLE II.

Group B was tested for both cracking and irreversible growth under normal underground magazine storage conditions (about 25°C in summer and 4°C. in winter) for about one year. No cracking was observed. Without considering seasonal expansion and shrinkage, a net growth of the order of 0".001 to 0".002 was noted - although some of this may be ascribed to human error. Only one decided increase was noted, i.e. Comp B containing cyclohexanone grew by about 0".006. Comp B containing catechol turned black.

Group C was tested for both irreversible growth and cracking by cycling between those magazine temperatures already stated and an oven temperature of 70° -74°C. Those slices of Comp B which contained alpha-nitronaphthalene, 4,6-dinitro-o-cresol, and catechol crumbled on the 3rd, 4th, and 6th cycles, respectively; hence do not appear in Figure 1 nor in TABLE II, which summarize the work done on Group C. Specifically, after being left in the magazine all night the explosive slices were removed from their box, their thickness and diameter measured, then placed in an oven at room temperature. The temperature of the oven was then brought to 70° -74°C in the course of three quarters of an hour to one hour's time, and the explosive left at that temperature all day. At the end of the day the slices were taken out, protected by wood shavings or paper bags, replaced in their box, and returned to the magazine. The next morning they were remeasured and any increase in size noted. This constituted one cycle. Under these conditions all the slices, except one, developed minor cracks during the first cycle. Some of these cracks were almost invisible, and the slice containing ortho-nitrotoluene as the additive had no visible cracks at all until the third cycle. Cracks always appeared on the flat surface and almost invariably started from the center, or "hub", and grew, as the number of cycles increased, toward the periphery of the slice. These cracks were always deep enough to reach the opposite surface. Some new cracks, or "radii", would develop as the cycling progressed, so that ultimately, when the charge finally broke, it was always into two, three, four, and sometimes five clean pieces, or sections. Since cracks seldom appeared on the curved surface of the charge - that is, cracks that might run along a plane parallel to the flat surface - it was always possible to continue measuring the growth in thickness of the charge by measuring the thickness of the section, even though the charge proper no longer existed. This measuring was continued until the section disintegrated by further cycling.

In Figure 1, the solid lines represent the growth of the cracked but unbroken charges. The measurements of the sections, representing only the growth in thickness of the explosive, is shown by the broken lines. The figures above either line represent the irreversible growth in thickness, while those

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below the line represent irreversible growth in diameter, both expressed in units of 1/1000 of an inch. In connection with diameter measurements it should be noted that often a crack, say, bisecting a charge, would tend to widen as the cycling proceeded. This widening was, however, always greater toward the center, or the middle, of the crack than at its tail ends, where it appeared pinched shut. This being the case, the measurement taken along, or parallel to, the crack was always smaller than the one taken at right angles to it. Due to this idiosyncrasy two or more diameter readings were taken, around the charge, and these averaged. The same was done for every charge. The diameters reported in Figure 1 are, therefore, only average figures. Since cracking did not occur along the curved surface, thickness readings are truer indices of the actual irreversible growth of the explosive and are, therefore, the ones especially considered herein.

It might be noted here that, since our Comp B control (no additive) fell by the wayside at the very beginning of these tests (see Column II, TABLE I), Comp B containing the additive ortho- and para-nitrotoluene (ONT/PNT ; .50% mixture) was taken as the new control, or point of reference, for the work that followed (see line 5, Figure 1). This was chosen because of its general popularity and use, and because it is the one anti-cracking agent most relied upon at this activity. In Figure 1 the additives are, on the whole, arranged in the order of increasing general efficiency. For instance, Comp B containing 2,4-dinitroanisole (line 1) fell apart (into sections) on the 14th cycle, whereas that containing cyclohexanone (line 2) did not fall apart until the 21st cycle. Again, Comp B containing 2,4-dinitrotoluene (line 3) had, on the 53rd cycle, increased in thickness by 0".068, and might have been a better anti-growth agent than ortho-nitrophenol (line 8), which caused an increase of 0".076 in the same number of cycles, had it not fallen apart on the 28th cycle. As stated above, the additive ONT/PNT (line 5) is now the new point of reference. This, together with ortho-nitrotoluene (line 4) show that Comp B in which they are contained fell apart on the 31st cycle. One might argue that para-nitrotoluene (line 6), showing a longer life without disintegrating as well as showing a smaller growth than lines 4 and 5, should be used alone, without mixing it with ortho-nitrotoluene. That this is not done is perhaps because, when used separately, Comp B containing para-nitrotoluene shows cracks, generally, much more readily (during the first cycle), whereas that containing ortho-nitrotoluene showed no cracks until the third cycle. By mixing the two additives together what is believed an acceptable compromise is reached. This type of compromise is well known by those interested. Comp B containing para-methoxybenzyl alcohol (line 7) shows roughly the same properties shown by that containing para-nitrotoluene,

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except that the growth in thickness, as well as in the diameter, are a little larger than in the latter case. Comp B containing ortho-nitrophenol (line 8) did not fall apart until the 53rd cycle. On the 31st cycle, when Comp B containing ONT/PNT (the new control) failed after attaining a growth in thickness of 0".028, that containing ortho-nitrophenol had grown only 0".017; and their corresponding average diameter had grown by 0".048 and 0".022, respectively. This difference in the two additives remained apparent all along, as can be seen on the 14th, 21st, 28th, and 31st cycles.

Figure 2 compares the control (ONT/PNT) with the best new additive (ortho-nitrophenol). In this, as in Figure 1, the solid part of curve (5) represents the growth in thickness of the cracked but unbroken Comp B containing ONT/PNT, while its broken continuation represents the growth in thickness of a section of the same Comp B slice. Curve (8) represents growth in thickness of Comp B containing ortho-nitrophenol as the additive. Figure 2 shows that:

- 1) The maximum growth of the first is 0".098, while that of the second is 0".076.
- 2) The first broke (fell apart) on the 31st cycle; the second on the 53rd.
- 3) On the 31st cycle the first had grown 0".028, while the second 0".017.

TABLE II shows, numerically and percentagewise, those considerations presented in Figure 1, in addition to some data on exudation and cracking. It will be noted that the more important data is found in columns II, III, IV, VIII, and IX. In column I the additives are arranged, as in Figure 1, in an approximate order of increasing efficiency. Column II shows the number of complete cycles withstood by the particular additive-containing Comp B slice without falling apart. In column III is shown the total irreversible growth in thickness, on the 31st cycle, of all those samples that survived until this cycle. As mentioned before, the 31st cycle was chosen because the new control, or point of reference, is Comp B which contains ONT/PNT and which disintegrated on the 31 cycle. The values are all in 1/1000 of an inch. The corresponding percentages, in parentheses, give an instant idea of the growth involved. Observe, how, except for the figures representing para-methoxybenzyl alcohol, which break the order, all figures in column III (and also IV) decrease as the figures in column II increase. Column IV shows the growth in diameter of the Comp B slice. For reasons already stated above, these are averages of two or more diameter measurements

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made around the particular slice. Since part of this growth is due to wide cracks, and since the width of these cracks might have been modified by a containing vessel, such as a shell, had they been so contained, the values in this column might have been other than what they are. For this reason thickness measurements, rather than diameter measurements, are to be considered the better growth representatives. Column V shows the total growth in thickness of the explosive samples on the 53rd cycle. Except for Comp B containing ortho-nitrophenol, which remained intact till this cycle, all the measurements in this column were taken from a section of the corresponding slices. It further shows that 2,4-dinitrotoluene would have been the best anti-growth agent of all had the Comp B containing it not fallen apart on the 28th cycle. Though the information on exudation (Column VIII) is not quantitative, since it was gleaned in passing, it is very much representative of the state of the explosive throughout the course of the experiment; and anyone conversant with the amount of exudation caused by para-nitrotoluene (LOW) and ortho-nitrotoluene (HIGH) can gauge the values of the rest. These values, qualitatively shown here, suffice to indicate the relative position of the various additives. A truly quantitative accounting of the cracks present in a given piece of cast explosive is a feat difficult to achieve. Perhaps a better measure of their number may be found in the results they effect; and something like this is shown in column II. Column IX, therefore, shows only those cracks which appear critical by reason of their width and length, but which, as it turned out, could not always be correlated with the data in column II. There is, for instance, good correlation between the number of major cracks and the ease with which the slice fell apart in the first four or five entries. Here the number of cycles in which they remain intact increase (from 14 to 31 cycles) as the number of major cracks decrease (from 8 to 3, respectively), but this convenient continuity does not hold for the rest of the entries. The indication that such an inverse correlation exists, however, appears unmistakable, and could be further corroborated by an examination of those other charges that perished early in the experiment and, consequently, do not appear in TABLE II.

Microscopic Examination of Comp B.

The action of the additive, or eutectic, appears limited to the regions (or interstices) between the crystals. That is, only the strength of the "cement" holding the crystals together, if one may so picture it, appears to be undermined by cycling. The individual crystals appear to remain the same size whether cycled or not. Cycling does not appear to make the individual crystals grow, such as at the expense of neighboring crystals.

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Sometimes the eutectic alone, other times cycling in the presence of some eutectics, appears to cause preferential alignment of the crystals, causing them to point either in a vertical direction along the charge, or in a horizontal one.

The broken surface of cycled Comp B is rougher and more granular than the broken surface of non-cycled Comp B. Non-cycled Comp B is smoother, more homogeneous in texture, and looks wet (like crystals soppy with mother liquor), while cycled Comp B looks dry, like crystals from which all surrounding liquid had been drained.

The "binding" material, or eutectic, which darkens on cycling, appears to flow between the crystals, away from some areas and on to certain others. Some spots in cycled Comp B are white and seem to have been drained of the binding material, which goes to darken other parts of the charge (dark amber).

SUMMARY

1) Tests have shown that, when used in large casts of Comp B, those additives usually reported as being satisfactory to good anticracking and anti-growth agents have almost totally failed when exposed to the rather severe conditions herein reported. Of these, only Comp B containing ortho-nitrotoluene and/or para-nitrotoluene survived cycling to the 31st and 32nd cycles, respectively.

2) Ortho-nitrophenol, when used as an additive in 5"x 8" cylindrical casts of Comp B, under the same severe conditions as above, was found to be a better anti-cracking and anti-growth agent than anything yet tried or reported, and the exudation resulting from it was perhaps the very minimum that can be expected from any additive.

3) Cracks developed vertically along the height of a cylindrical charge (forming like radii on its flat surface) much more easily than they did horizontally.

4) The additive-containing Comp B that cracks first, or that has the most cracks, does not always fall apart first, although the trend is in that direction.

5) Under normal, underground magazine storage conditions, Comp B charges appear not to grow appreciably (on the basis of 1 year's surveillance).

RECOMMENDATIONS

It is recommended that the following work be undertaken so as to complete the present study:

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1) Quantitative tests to determine the actual amount of exudate formed when ortho-nitrophenol is used as an additive for Comp B, using as controls Comp B containing ortho-nitrotoluene and para-nitrotoluene, separately, or any other appropriate control.

2) Try, as crack preventing and growth preventing additives, compounds having the following characteristics:- aromatic nucleus containing any two of the following groups, in the ortho position: Nitro, Hydroxyl, Methyl, and Methoxy. The compound in question to be low melting, preferably below 80°C.

3) Try, as an additive for the same purpose, the eutectic formed by combining para-methoxybenzyl alcohol and para-nitrotoluene.

TABLE I
CRACKING PROPENSITY OF COMP B CONTAINING VARIOUS ADDITIVES

I ADDITIVE IN COMPOSITION B (0.5%)	II Cracked on cooling to room temp. Sudden change from 83° to 24°C. (X-Rayed)	III Cracked during 1st Temp.Shock. Sudden change from 0° to 24°C. (X-Rayed)	IV Cracked during 2nd Temp. Shock. Sudden change from 0° to 24°C. (X-Rayed)	V Cracked during 3rd Temp. Shock. Sudden change from 0° to 60°C. (X-Rayed)
NONE	Completely			
Nitrocellulose	Completely			Very badly
Shawinigan Black	Completely			Very badly
Pyrogalllic Acid	Completely			Very badly
Anthracene	Completely			Badly
Sorbitol Monooleate	Completely			Badly
Biphenyl	Completely			Badly
2,4,6-Trinitroanisole	Completely			Not badly
4,6-Dinitro-o-cresol	Completely			Not badly
2,4-Dinitroanisole	Completely			Not badly
Catechol		None	Slightly	Not badly
Cyclohexanone		None	None	Not badly
Alpha-Nitronaphthalene		None	None	Not badly
2,4-Dinitrotoluene		None	Slightly	Not badly
p-Methoxybenzyl Alcohol		Slightly	None	Not badly
p-Nitrotoluene		None	None	Not badly
o-Nitrotoluene		None	None	Not badly
ONT/PNT (50/50)		None	None	Not badly
o-Nitrophenol		None	None	Not badly

TABLE II
IRREVERSIBLE GROWTH OF COMP B CONTAINING VARIOUS ADDITIVES

I ADDITIVE IN COMP B	II No. of cycles before break- ing	III		IV		V		VI		VII		VIII		IX No. of major, or critical, cracks
		Total growth in thick- ness on 31st cycle	(in 0".001) (in 0".001)	Total growth in diameter on 31st cycle (average)	(in 0".001) (in 0".001)	Total growth in thick- ness on 53rd cycle	(in 0".001) (in 0".001)	Total growth in thick- ness on breaking	(in 0".001) (in 0".001)	Total growth in diameter on break- ing (average)	(in 0".001) (in 0".001)	Exudation (Qualitative)		
2,4-Dinitroanisole	14							9 (0.35%)	19 (0.38%)			Low	8	
Cyclohexanone	21							41 (1.63%)	59 (2.35%)			High	6	
2,4-Dinitrotoluene	28					68 (2.68%)		15 (0.59%)	22 (0.44%)			Low	4	
o-Nitrotoluene	31	34 (1.32%)	62 (1.24%)	48 (0.96%)	106 (4.16%)	34 (1.32%)	62 (1.24%)	34 (1.32%)	62 (1.24%)			High	3	
ONT/PNT *	31	28 (1.09%)	48 (0.96%)	98 (3.84%)	28 (1.09%)	28 (1.09%)	48 (0.96%)	28 (1.09%)	48 (0.96%)			High	3	
p-Nitrotoluene	32	20 (0.78%)	35 (0.71%)	85 (3.34%)	23 (0.90%)	23 (0.90%)	39 (0.78%)	23 (0.90%)	39 (0.78%)			Low	3	
p-Methoxybenzyl alcohol	34	25 (0.98%)	46 (0.92%)	78 (3.07%)	31 (1.22%)	31 (1.22%)	62 (1.24%)	31 (1.22%)	62 (1.24%)			Medium	4	
o-Nitrophenol	53	17 (0.66%)	22 (0.44%)	76 (2.98%)	76 (2.98%)	76 (2.98%)	107 (2.14%)	76 (2.98%)	107 (2.14%)			Low	3	
*														
o-Nitrotoluene and p-Nitrotoluene														

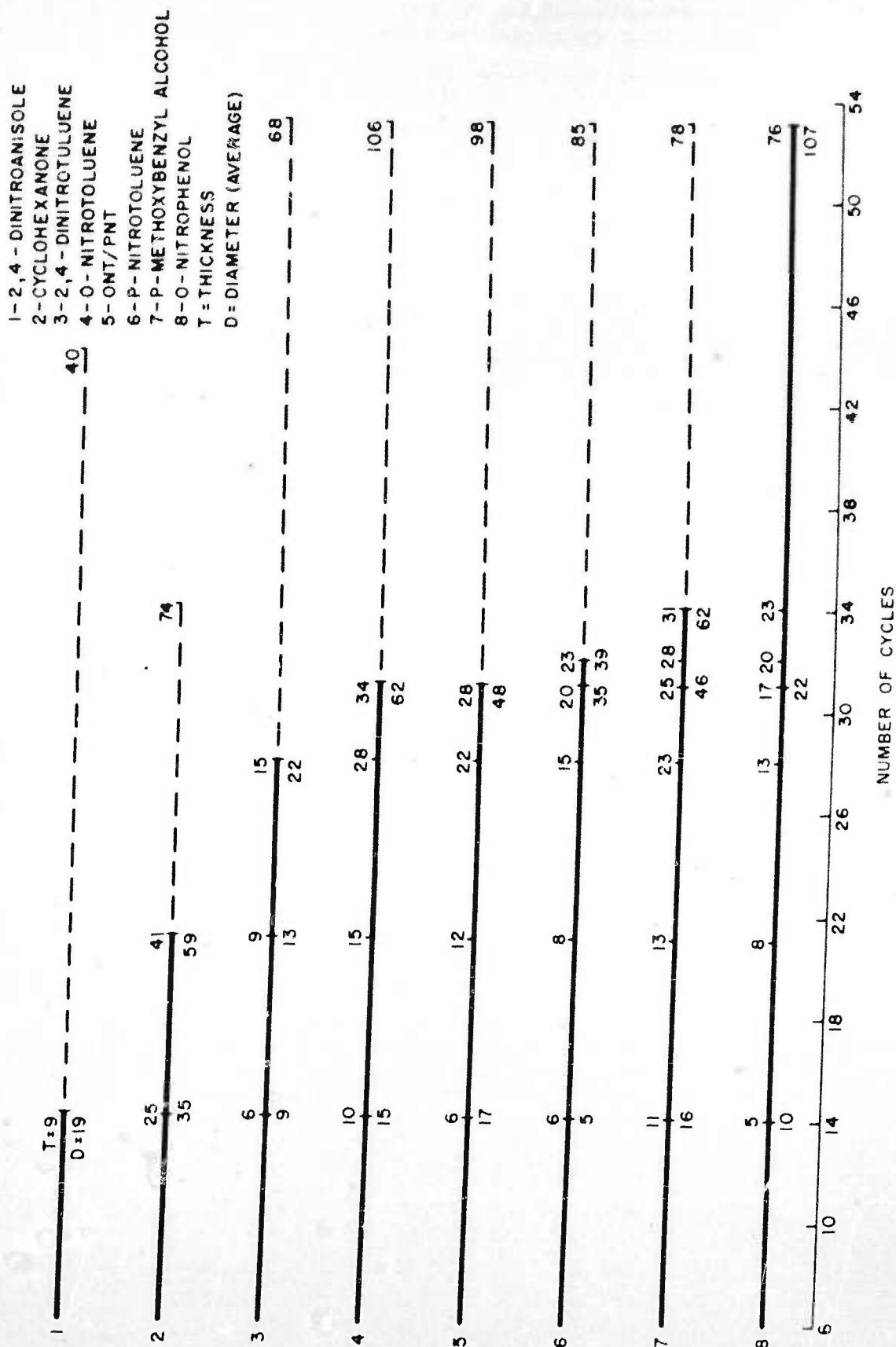


FIG. 1 RELATIVE EFFICIENCY OF VARIOUS ADDITIVES AS ANTICRACKING AND
ANTIGROWTH AGENTS IN COMP B

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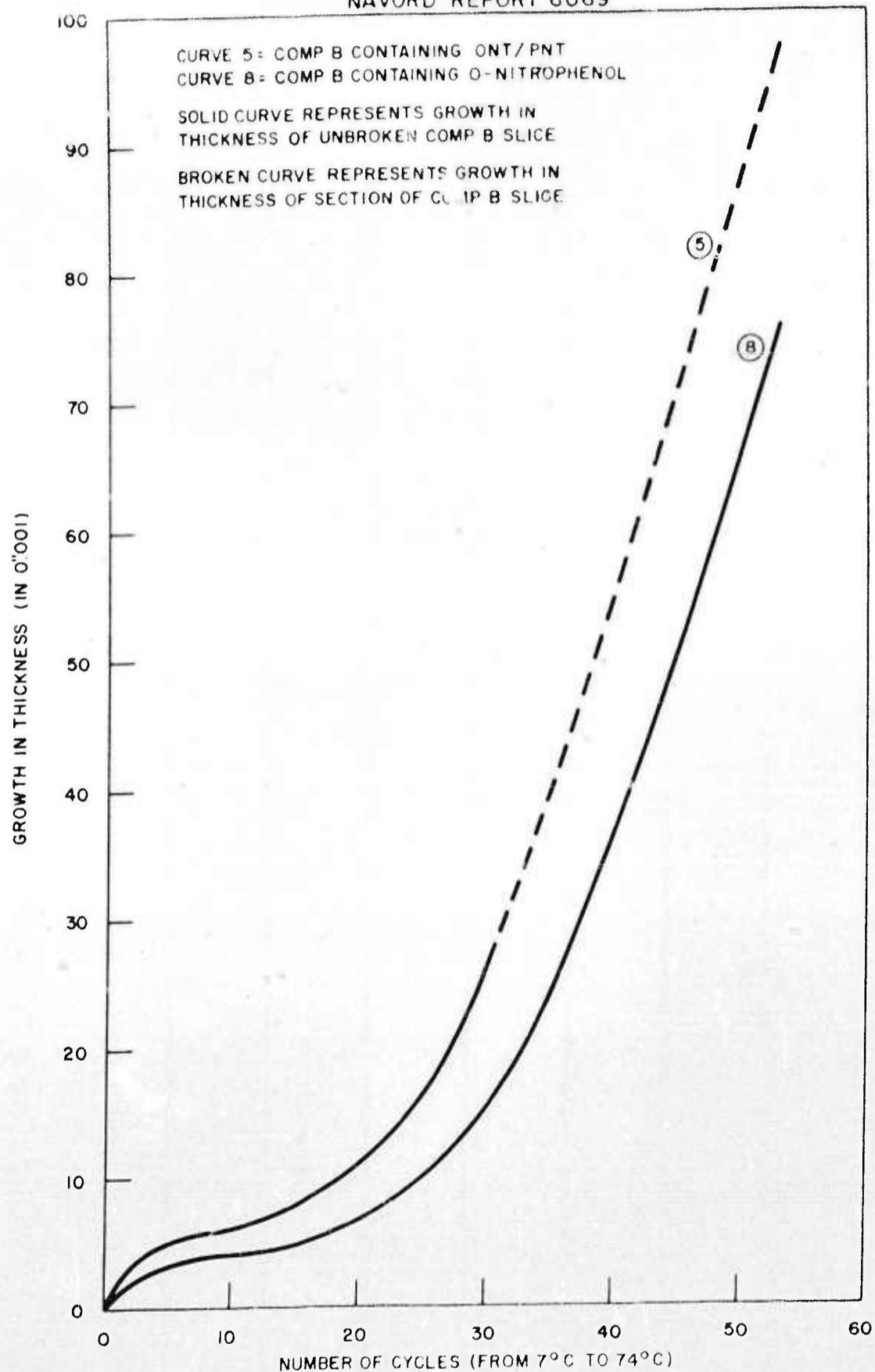


FIG. 2 RELATIVE EFFICIENCY OF O-NITROPHENOL AND ONT/PNT AS ANTICRACKING AND ANTIGROWTH AGENTS IN COMP B

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